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XXIII. *Experiments and Observations on the Terra Ponderosa, &c. By William Withering, M. D.; communicated by Richard Kirwan, Esq. F. R. S.*

Read April 22, 1784.

## S E C T I O N I.

### *Terra ponderosa aërata.*

THIS substance was got out of a lead-mine at Alston-Moor, in Cumberland. I first saw it in the valuable collection of my worthy and ingenious friend MATTHEW BOULTON, Esq. at Soho; who, when he picked it up, conjectured from its weight that it contained something metallic. About two years ago I saw it in his possession; and partly from its appearance, being different from that of any calcareous spar I had seen, and partly from its great weight, I suspected it to be the spatum ponderosum.

A few experiments made at the moment confirmed my suspicions, at least so far as to shew that it contained a large proportion of the terra ponderosa united to fixed air; but I did not then flatter myself that it would prove so pure as I afterwards found it to be.

Professor

Professor BERGMAN, in his *Sciagraphia Regni Mineralis*, published last year at Leipzig, conjectures (§ 58.), with his usual sagacity, “*Terra ponderosa nitrata forte alicubi nativa occurrit, a nemine tamen adhuc inventa, quod etiam valet de aërata.*”

I was much delighted by the detection of a substance which promises to be of very considerable utility in chemical inquiries, and more so when I found it to be a native of this country; for it is not improbable, that it may be met with in many other mines, besides that at Alston-Moor.

Mr. BOULTON, with his usual benevolence, presented me with a piece of it, part of which accompanies this paper, for the inspection of the Members of the Royal Society.

### *More obvious Properties.*

Its general appearance is not much unlike that of a lump of alum; but, upon closer inspection, it seems to be composed of slender spiculæ in close contact, but more or less diverging. It may be cut with a knife. Its specific gravity is from 4,300 to 4,338.

It effervesces with acids, and melts under the blow-pipe, though not very readily. Placed in a covered crucible, in a hot parlour fire, it lost its transparency.

After exposure to a moderate heat in a melting furnace, it adhered to the crucible, and exhibited signs of fusion; but was not diminished in weight, did not feel caustic when applied to the tongue, nor had it lost its property of effervescing with acids.

Hence

Hence it is probable, that its loss of transparency was rather occasioned by numerous small cracks, than by any escape of the water of crystallization, or of its aerial acid.

## E X P E R I M E N T S.

A. 500 grains, dissolved in muriatic acid, in such a manner that nothing but elastic fluid could escape, lost in solution 104 grains, and there remained an insoluble residuum of nearly 3 grains.

2. In another experiment 100 grains lost in solution 21 grains, and there remained 0,6 of a grain of insoluble matter.

B. 100 grains dissolving in dilute muriatic acid, gave out 25 ounce measures of air. This air was received in quicksilver, and when the spar was wholly dissolved, the solution was boiled, in order to drive out what air might be lodged in it.

2 This air was heavier than atmospheric air; it was readily absorbed by agitation in water, it precipitated lime from lime-water, and it extinguished flame. The water which had absorbed it changed the blue colour of litmus slowly \* to a red; so that this elastic fluid was undoubtedly fixed air.

C. The solution (B) by the addition of mild fossil fixed alkaly, afforded a precipitate which, after proper washing and drying, weighed 100 grains.

2. This precipitate, upon being again dissolved in marine acid, yielded only 20 ounce measures of fixed air.

\* Other acids turn the blue of litmus instantly to a red, whilst water, impregnated with fixed air, does not change the litmus immediately; but, after some seconds, the red colour begins to appear, and then gradually grows more distinct.

D. To a saturated solution in marine acid mild fixed vegetable alkali was added; the earth was precipitated, and a quantity of fixed air escaped.

2. The same thing happened when mild fossil alkali was added.

3. When caustic vegetable alkali was used, the precipitation took place, but without any appearance of effervescence.

4. 50 parts dissolved in marine acid lost, during the solution, nearly 10,5. This solution, upon the addition of caustic vegetable alkali, let fall a precipitate which, when washed and dried, weighed 45,5.

5. Phlogisticated alkali precipitated the whole of the earth from part of the solution D; for mild fixed alkali afterwards added to the filtered liquor occasioned no further precipitation.

E. Part of the precipitates D. 1. 2. after exposure to a strong heat in a crucible, was thrown into water. Next morning the water was completely covered with an ice-like crust, and had the acrid taste of lime-water in a very high degree.

2. The smallest portion of vitriolic acid added to this water occasioned an immediate and copious precipitation; and when this acrid water was diluted with 200 times its bulk of pure water, the precipitation upon the addition of vitriolic acid was yet sufficiently obvious.

3. A single drop of this acrid water, added to solutions of tartar of vitriol, GLAUBER's salt, vitriolic ammoniac, alum, Epsom salt, felenite, occasioned an immediate precipitation in all of them.

F. The precipitate thrown down by the caustic vegetable alkali (D. 3.) was put into water, in expectation that it would make lime-water; but neither upon standing, nor after boiling, did this water exhibit any precipitation when concentrated  
vitriolic

vitriolic acid was dropped in it; nor had it any acrimonious or other peculiar taste.

G. Concentrated vitriolic acid was added to one portion of the precipitate D. 3; concentrated nitrous acid to a second portion; and marine acid to a third portion. No effervescence could be observed, nor was there any appearance of solution. After standing one hour water was added; and the acids, thus diluted, were suffered to remain upon the portions of the precipitate for another hour. They were then decanted, and saturated with mild fossil fixed alkaly, but without any appearance of precipitation.

H. The part precipitated by the phlogificated alkaly, when mixed with nitre and borax, and fluxed by a blow-pipe upon charcoal, formed a black glass; upon flint-glass, a white; and upon a tobacco-pipe an opaque yellowish white one.

2. Another portion melted with soap and borax in a crucible, formed a black glass, but without any metallic appearance.

I. The insoluble residuum (A.) was boiled in water, the water decanted, and mild fixed alkaly added, but without any subsequent precipitation.

2. This insoluble powder was not attacked by the nitrous or marine acids; but being put into vitriolic acid, and boiled a considerable time until the acid became highly concentrated, it dissolved entirely, and separated again upon the addition of water. It will appear in the sequel, that the same thing happens to marmor metallicum, when dissolved by boiling in the acid of vitriol.

## C O N C L U S I O N S.

Hence it appears, that 100 parts of this spar contain

Terra ponderosa pura	-	78,6
Marmor metallicum	-	,6
Fixed air	- - -	20,8
		<hr/>
		100.
		<hr/>

## O B S E R V A T I O N S.

1st, The quantity of mild fixed alkaly necessary to saturate an acid, previously united to the terra ponderosa, contains more fixed air than is necessary to saturate that quantity of terra ponderosa D. 1. 2.

2dly, The terra ponderosa, when precipitated from an acid by means of a mild fixed alkaly (D. 1. 2.), readily burns to lime; and this lime-water proves a very nice test of the presence of vitriolic acid. E. 2. 3.

3dly, It is very remarkable, that the terra ponderosa spar, in its native state, will not burn to lime. In the lower degrees of heat it suffers no change, as was before observed, besides the loss of its transparency. When urged with a stronger fire, it melts and unites to the crucible, but does not become caustic.

I buried it in charcoal-duft in a covered crucible, and then exposed it to a pretty strong heat; but it did not part with its air.

May we not conjecture, then, that as caustic lime cannot unite to fixed air without the intervention of moisture, and as this spar seems to contain no water in its composition, that it

is

is the want of water which prevents the fixed air assuming its elastic aërial state? This supposition becomes still more probable, if we observe that when the solution of the spar in an acid is precipitated by a mild alkaly, C. 1. 2. some water enters into the composition of the precipitate, for it weighs the same as before it was dissolved, and yet contains only 20 ounce measures of fixed air, whilst the native spar contained 25 ounce measures; so that there is an addition of weight equal to that of 5 ounce measures of fixable air, or  $3\frac{1}{4}$  grains to be accounted for, which can only arise from the water; and this precipitate, thus united to water, readily loses its aërial acid in the fire, E. 1.

4thly, Professor BERGMAN supposes the terra ponderosa to be a metallic earth\*; its entire separation from an acid by means of the phlogisticated alkaly (D. 5.) certainly favours such a supposition; but, if it be so, it is evident from experiments H. 1. 2. that other means than those commonly employed must be used to effect its reduction.

5thly, The precipitate made by the caustic vegetable alkaly D. 4. taking some of the alkaly down with it, and thus forming a substance neither soluble in water nor in acids, is a very curious phænomenon.

I afterwards varied the experiment by adding the terra ponderosa lime-water (E.) to caustic vegetable and caustic fossil alkaly. In both cases this insoluble compound was immediately formed; but not so when caustic volatile alkaly was used. This composition of an alkaly and an earth certainly deserves more attention than I am at present able to bestow upon it.

6thly, As it appears from experiments D. 1. 2. 3. 4. that

\* See preface to his *Sciagraphia Regni Mineralis*.



fixed alkalies, both mild and caustic, separated the terra ponderosa from marine acid, I was at a loss to know why Professor BERGMAN, in his admirable table of simple elective attractions, placed the terra ponderosa caustica immediately under the vitriolic, nitrous, and marine acids; and consequently above the caustic alkalies. I was interested in the reality of the facts, because I had so seldom seen reason to doubt the observations of that very excellent chemist, and therefore made the following experiments.

To different portions of terra ponderosa salita and terra ponderosa nitrata I added, drop by drop, caustic vegetable, caustic fossil, and caustic volatile alkalies. In every case the EARTH was thrown down; and I have so often repeated these experiments to satisfy myself and others, that I am persuaded the terra ponderosa caustica ought to be placed below the alkalies, except in the column appropriated to the vitriolic acid; and it may be separated even from that acid, by the vegetable fixed alkaly, if the alkaly be applied *via sicca*, as will appear in the next section.

7thly, The necessity for using pure acids upon many occasions, and the difficulty of obtaining them pure, are sufficiently obvious. The VITRIOLIC ACID, as made in the large way, is generally pure enough for most purposes. It is apt to get coloured by inflammable matter; but this is seldom an inconvenience; and, when it would be so, it is easy to drive it off by boiling the acid in a Florence flask over a common fire. But there is another cause of impurity in this acid, which appears upon diluting it with water; for then it becomes milky, and in a short time a powder subsides\*.

The

\* About two years ago I examined this powdery matter; both that which was thrown

The acid may be freed from this powder either by distillation in glass vessels, which is a tedious and dangerous process, or by the affusion of water; and, after the powder has subsided, a gentle evaporation will drive off most of the superfluous water.

NITROUS ACID may be freed from vitriolic and marine acids, by solution of silver in the acid of nitre, as is daily practised; but the MARINE ACID has not, to my knowledge, been purified by any other method than the laborious one of re-distilling it from common salt. It is generally mixed with vitriolic acid; and often in large proportion. There is no temptation, and scarcely an opportunity, for it to be contaminated by nitrous acid. From the vitriolic acid then it may be readily purified by the addition of terra ponderosa caustica dissolved in water, or by the terra ponderosa salita. If the latter be used, a small

thrown down by dilution with water, and also some which Dr. PRIESTLEY gave me, being the residuum of vitriolic acid distilled to dryness in a flint-glass retort.

1st, Repeated boiling in water, reduced  $6\frac{1}{2}$  grains to 2 grains.

2dly, This solution, by gentle evaporation, afforded 5 grains of crystals, as hard and as tasteless as selenite.

3dly, To these crystals, re-dissolved in water, mild fossil alkaly was added, and a white powder precipitated.

4thly, This powder, after exposure to a pretty sharp heat, was thrown into water; part of it dissolved, and the water got the taste and other properties of lime-water.

5thly, The insoluble part (1.) suffered no change by boiling in nitrous acid; one-half of it mixed with borax, and exposed to the blow-pipe upon charcoal, vitrified; the other half, mixed with borax and charcoal-dust, likewise vitrified.

CONCLUSIONS. It appears, then, that the greater part of this substance was calx vitriolata, or selenite; the remainder a vitrifiable earth.

I had before found, that the terra ponderosa vitriolata, or heavy gypsum, would dissolve in concentrated vitriolic acid; but always separated in a powdery form upon the affusion of water; and now it appears, that calx vitriolata, or selenite, does the same.

portion of the acid must first be tried in a diluted state, from whence we must judge how much of the terra ponderosa salita the whole will require; or else the whole of the acid must be diluted with water. Whether we use the terra ponderosa dissolved in water or in marine acid; in either case the acid of vitriol immediately seizes upon it, and subsides with it in form of an insoluble powder.

As there are reasons for preferring the marine acid in several of the nicer and more important enquiries of chemistry, this ready method of purifying it cannot but prove acceptable.

## S E C T I O N II.

Terra ponderosa vitriolata. BERGMAN's Sciagraphia, §§ 58. 89.

*Variety*, Heavy Gypsum. Ponderous Spar.

Marmor Metallicum. CRONSTEDT Min. § 18. 2. 19. C.

From Kilpatrick-hills near Glasgow. A fort, with smaller crystals, amongst the iron ore about Ketley in Shropshire. In the lead mines at Alston-Moor.

### *More obvious Properties.*

White; nearly transparent, but has not the property of double refraction; composed of laminæ of rhomboidal crystals; decrepitates in the fire. Specific gravity from 4,402 to 4,440.

## E X P E R I M E N T S.

A. 100 grains exposed to a red heat for one hour, in a black lead crucible, lost five grains in weight; but as a sulphureous smell was perceptible, I suspected that a decomposition had taken place, and therefore exposed another portion to a similar heat for the same space of time in a tobacco-pipe. This had no smell of sulphur, nor was it diminished in weight.

2. It is barely fusible under the blow-pipe; but with borax fluxes readily into a white opaque glass.

B. 100 grains, ground in a mortar, and washed over extremely fine by repeated additions of water, were boiled in the same water, and, after settling, the water was poured off. The powder, when dried, had not sensibly lost weight.

2. To separate portions of the washing water, were added mild vegetable and mild fossil alkaly; but without any appearance of precipitation. Nitre of mercury gave a very slight brownish cloud, barely discernible; and nitre of silver an extremely slight bluish appearance.

3. The same powder, boiled again in fresh water, did not affect the water at all; for it stood the test of nitre of silver without any change.

C. Portions of the powder B. were boiled in vitriolic, nitrous, and muriatic acids, of the usual strength, for several minutes. The acids were then saturated with vegetable fixed alkaly, but without any appearance of precipitation, nor had the portions of powder lost any weight.

2. But when boiled in vitriolic acid, until that acid became very much concentrated and nearly red-hot, the whole of it dissolved; but, separated again upon the addition of water, was

not altered in its weight, was not acted upon by acids of the usual strength, and had, under the blow-pipe, the properties mentioned at A. 2.

3. Some of the solution in the concentrated vitriolic acid was left exposed to the atmosphere, that the acid might slowly attract water. After some days, beautiful crystals appeared in the shapes of stars, fasciæ, and other radiated forms.

4. To another portion of this solution mild fixed vegetable alkaly was added; but the precipitate appeared to be the marmor metallicum unchanged.

D. One ounce of this marmor metallicum in fine powder was fluxed in a crucible with two ounces of salt of tartar, until it ran thin. This substance, boiled with water in a Florence flask, left a residuum of six drams.

E. This residuum was thrown into water, and pure nitrous acid added, until there was no more effervescence. The undissolved part weighed 52 grains.

F. This undissolved part appeared to be the original substance no ways changed; for it did not dissolve in nitrous or marine acids, but did wholly dissolve in the greatly concentrated and boiling vitriolic acid, from which it was again separated by the addition of water. (C. 2.)

G. The solution D. was saturated with distilled vinegar, and then evaporated to dryness, but with less than a boiling heat. The sal diureticus, thus formed, was washed away with alcohol. The remaining salt weighed 5 drams nearly.

2. This salt had the appearance and the taste of vitriolated tartar; it decrepitated in the fire; roasted with charcoal dust, it formed a hepar sulphuris; and with muria calcarea gave a precipitation of selenite.

H. The salt, formed with the nitrous acid (E), shot readily into beautiful permanent crystals, of a rough bitterish taste.

2. Some of this salt was deflagrated with nitre and charcoal, and the alkaly afterwards washed away.

3. The residuum, being the earth of the marmor metallicum, was very white, burnt to lime, and again formed an insoluble compound with acid of vitriol.

I. 100 grains of terra ponderosa aërata were dissolved in diluted marine acid. Vitriolic acid was dropped into this solution, until no more precipitation ensued. The precipitate, after very careful washing and drying, was exposed to a red heat in a covered tobacco-pipe for half an hour: when cool, it weighed 117 grains.

2. 50 grains of terra ponderosa aërata in a lump were put into diluted vitriolic acid; but the action of the acid upon it was hardly sensible, even when made hot.

Marine acid was then added, and after some time an effervescence appeared. The terra ponderosa vitriolata, thus formed, after proper washing and drying, was exposed to a red heat for an hour: it then weighed 58,4 grains.

#### CONCLUSIONS.

1st, It appears that the marmor metallicum is composed of vitriolic acid and terra ponderosa, D. E. F. G. H.

2dly, That this compound, though probably soluble in water, has so little solubility as almost to escape detection by the nicest chemical tests, B. 1. 2. 3.

3dly, That it is not soluble in acids of the usual strength; but that it perfectly and entirely dissolves in highly concentrated

trated vitriolic acid, from which it again separates entire and unchanged upon the affusion of water, C. 1. 2.

4thly, That it cannot be decomposed (*via humida*) by mild fixed alkaly, C. 4.

5thly, That it may be decomposed (*via sicca*) by the vegetable fixed alkaly, D. E. G. H.

6thly, That it may be decomposed by inflammable matter, uniting to its acid, and forming sulphur; but that it cannot be decomposed by heat alone, A. 1.

7thly, From experiments I. 1. 2. it appears, that 100 parts of this substance contain

Vitriolic acid pure	-	32,8
Terra ponderosa pure		67,2
		<hr/>
		100.

For the 100 parts of terra ponderosa aërata made use of in the experiment I. 1. would lose during the solution 20,8 of fixed air (§ 1st, A.); then, deducting 0,6 for the marmor metallicum contained in the terra ponderosa aërata (§ 1st. A. 1. 2.), there remains 78,6 of pure terra ponderosa. This, when saturated with vitriolic acid, and made perfectly dry, weighed 117; consequently it had taken 38,4 of vitriolic acid.

#### OBSERVATIONS.

The apparent insolubility of terra ponderosa aërata in the diluted vitriolic acid (I. 2.) can be accounted for by remarking, that the moment the surface of the lump was acted upon by the acid, an insoluble coat of marmor metallicum was formed upon it, which effectually precluded any further operation of the acid.

Professor

Professor BERGMAN, in order to obtain the earth from the terra ponderosa vitriolata, directs the latter to be roasted with fixed alkaly, and the dust of charcoal; but I have always done it by charcoal dust alone, though probably this method may require a greater degree of heat.

It has been remarked, that terra ponderosa and calx of lead resemble each other in many respects; and I must add, that the vitriol of lead dissolves in the highly concentrated vitriolic acid much in the same manner that the marmor metallicum does, and like this too separates upon the affusion of water; but I never observed it disposed to crystallize.

The marmor metallicum may probably be useful in some cases where a powerful flux is wanted; for I once mixed some of it with the black flux, and exposing it to a pretty sharp heat, it entirely ran through the crucible. May not, therefore, some of the more common varieties of it be used advantageously as a flux to some of the more refractory metallic ores?

### S E C T I O N III.

Terra ponderosa Vitriolata.

*Variety, Calx or Cauk.*

Marmor Metallicum, CRONSTEDT Min. § 18. B?

Plentiful in the Mines in Derbyshire.

#### *More obvious Properties.*

Of a white or reddish colour; crystallized in rhomboidal laminæ, but these very much intermixed and confused. Loies



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little or nothing of its weight by being made red-hot. Specific gravity 4,330.

#### EXPERIMENTS.

A. Ground in a mortar, and washed over, the washing water, when decanted, gave no precipitation with mild vegetable alkaly; but with nitre of silver and nitre of mercury the slightest cloud imaginable.

B. 100 grains boiled in marine acid weighed, after proper washing and drying, 99,5.

C. The acid solution B let fall a Prussian blue upon the addition of a single drop of phlogisticated fixed alkaly; and, when saturated with mild fossil alkaly, afforded an ochry-coloured precipitate.

D. This precipitate, collected and washed, weighed half a grain. It was roasted with tallow, and then was wholly attracted by a magnet.

E. A quantity of the cauk, finely powdered, was mixed with charcoal-duft, and roasted in a crucible at a white heat, for five hours, fresh charcoal-duft being occasionally added. It gave out a strong smell of sulphur.

F. To this roasted cauk nitrous acid was added, which dissolved the greater part of it; producing, during the solution, some effervescence, and a strong smell of hepar sulphuris.

G. Some of this solution, after proper evaporation, afforded beautiful crystals, not deliquescent, exactly resembling those obtained from the marmor metallicum, (§ II. H.).

H. To other portions of the solution F, were added fixed vegetable and fossil alkalies, and likewise volatile alkaly, each of which precipitated the earth from the acid.

I. This

I. This earth, after exposure to a white heat for one hour, became caustic, and made lime-water, similar in properties to to that mentioned at § 1st. E.

K. Some of the part not acted upon by the nitrous acid F, dissolved entirely by boiling in highly concentrated vitriolic acid, and wholly separated again by the affusion of water. More water was added, and the whole was boiled again; but the filtered liquor gave no signs of precipitation upon the most liberal addition of mild fixed vegetable alkaly.

#### CONCLUSIONS.

It appears, therefore, that 100 parts of Derbyshire cauk contain

Marmor metallicum	-	99,5
Calciform iron	-	,5
		<hr/>
		100
		<hr/>

And it is probable, that the redder pieces contain a little more iron.

#### SECTION IV.

Terra ponderosa vitriolata.

*Variety*, radiated Cauc.

Gypsum crystallifatum capillare. CRONSTEDT Min

§ 19. B.

From Pennely by the Bog, near Minsterley, in Shropshire, fifteen miles from Salop, on the road to Montgomery.

*More obvious Properties.*

Somewhat glossy like fatin; yellowish-white, opaque; composed of slender spiculæ set close together, and pointing from a center.

In some pieces there are concentric circles of a semi-transparent horn like appearance. It is not very brittle; may be shaved with a knife; loses little or nothing of its weight by being made red-hot. Its specific gravity 4,000; but after soaking one night in the water 4,200, or more.

## E X P E R I M E N T S.

When treated in the same manner that the Derbyshire cauk was, in the preceding section, 100 parts of it appeared to contain

Marmor metallicum	-	97,7
Calci form iron	-	2,3
		<hr/>
		100

Suspecting that the presence of so small a proportion of iron could hardly occasion the whole of the apparent differences betwixt the Shropshire and Derbyshire cauks and the marmor metallicum; and thinking it not improbable, that they might contain lead; I mixed some of them with charcoal-dust and borax, but could not by means of the blow-pipe produce any metallic appearance, although vitriol of lead, treated in the same manner, was readily reduced.

I then mixed four parts of cauk with one part of vitriol of lead; the lead could still be reduced, though not so readily as before.

GENERAL OBSERVATIONS.

The terra ponderosa seems to claim a place betwixt the earths and the metallic calces. Like the former, it cannot be made to assume a metallic form; but, like the latter, it may be precipitated from an acid, by means of phlogisticated alkaly. In many of its properties it much resembles the calx of lead; and in others, the common calcareous earth, but still seems sufficiently different from that to constitute a new genus, as will appear from a little attention to the following circumstances.

Terra ponderosa,	Terra calcarea,
When dissolved in water, precipitates upon the addition of the smallest portion of vitriolic acid.	Dissolved in water, does not precipitate upon the addition of vitriolic acid.
Its gypsum, therefore, is insoluble.	Its gypsum, therefore, is soluble.
With the nitrous and marine acid, forms crystals which do not deliquesce.	With nitrous and marine acids forms salts so deliquescent that they cannot be kept in a crystallized form.
Decomposes vitriolic salts <i>via humida</i> .	Does not decompose vitriolic salts.

It has been called terra ponderosa, or heavy earth, upon account of the great specific gravity of its gypsum; its spar is likewise heavy enough to countenance such an appellation; but the earth itself does not appear to be a heavy substance, and I imagine the great weight of its compounds with the vitriolic and aerial acids is owing to the absence of water.

Birmingham, Nov. 1783.

